Wine volatiles composition in the sensory evaluation of bouquet and flavour of two vine cultivars

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ABSTRACT: Analytic measurement data acquired by an SFE/CGC analysis were compared with sensory evaluation of two wines from South Moravia. The sensory descriptive analysis showed a large variation in sensory quality from neutral character to full expression of the variety. Whereas the cluster analysis of volatiles was sufficient for differentiating both wines, the sensory observation data based on seven descriptors did not suffice either for bouquet or flavour characteristic. The descriptor citrus could be related to analytically established substances whose statistical significance was supported by a stepwise multiple analysis. The possibility of recognising vine cultivars via sensory descriptors is statistically feasible only by nutty and caramel descriptors in the case of bouquet and by big fruit for flavour.

Keywords: wine; sensory analysis; aroma compounds; supercritical fluid extraction/CGC analysis

Wine aroma is an important aspect of wine quality to which volatile components make an important contribution. Cultivars may differ substantially in viticultural performance and their potential to produce aroma-rich characteristics for yielding extracts and cultivar-typical wines (MARAIS, RAPP 1991; MARAIS 1994; BERNET et al. 1999; GUTH 1997; LÓPEZ et al. 1999; FERREIRA et al. 2000). When wines are produced, the most appropriate wine-making techniques for the maintenance of the typical cultivar characteristic have to be applied. Terpenes have been recognised for a long time as important components of grape and wine. The basis of sensory accessible varietal wine expression seems to be terpenoid compounds. On the basis of terpenes, pattern differences between Riesling and some of its descendants could be ascertained. Only six variables were necessary to achieve a reliable separation between two varieties and the agreement with sensory evaluation (RAPP et al. 1993). An analytical characterisation of neutral wine is describable by about 20 compounds (RAPP 1998). Sensory evaluation could detect significant differences by a triangle test in Chardonnay wine aroma due to malolactic fermentation (LAURENT et al. 1994). Flavour of wine in neutral non-aromatic varieties as Sylvaner type has only a low concentration of free monoterpenes (RAPP, GÜNTERT 1985; RAPP et al. 1993).

Gas chromatography with olfactometric detection has proved to be a powerful method to determine key compounds of food aroma (PRISER et al. 1997).

The commonly used methods for estimating volatile organic material from natural sources are steam distillation, solvent extraction, heat desorption or vapour collection by cryogenic concentration or adsorption to solid adsorbents (charcoal, silicagel, Tenax GC). Trace compounds that are present even in ultra-pure solvents act disturbingly on the analysis when working in the μl/l range.

The direct gas chromatographic analysis of aroma compounds over a liquid phase has several attractive features, e.g. only a small quantity of samples is usually required and, in addition, little sample preparation is necessary, thus keeping the formation of artefacts to a minimum. However, important aroma compounds are frequently present in the headspace vapour over the liquid phase at a concentration detectable by the human nose, but not by gas chromatographic or mass spectroscopic detectors.

The advantages of supercritical fluid extraction (SFE) over conventional liquid extraction methods follow from the properties of supercritical fluid which in moderate densities possesses sufficient dissolving power, lower viscosity, and higher solute diffusion potential (JANDA et al. 1996). SFE applications have so far been focussed mainly on solids, but a direct SFE of aqueous matrices like wine for continuous extraction in co-current mode has also been used (VEJROSTA et al. 1998).

Multivariate statistical techniques offer a powerful aid for the interpretation of chromatographic or sensory data. The stepwise discriminating analysis was used by SCHREIER et al. (1976) to determine substances that best separate different wine varieties and by MARAIS et al. (1981) to select components which relate to the geographic origins of wines.

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What sense organs are concerned in the detection of taste, odour and aroma? We regard taste as the property of liquids or solids and gases in solution that is detected in the mouth, not only by receptor cells in the taste buds of the tongue but also elsewhere in the oral cavity. Odour is designated when the volatile compounds follow the nasal passage way to the smell receptors as when wine is sniffed, and it is called aroma when the molecules reach the receptors by the retro-nasal passage as it must when the wine is in mouth. The synchronous sensation of taste and odour leads to the perception of flavour. Like in the case of most food products the aroma or "bouquet" of a wine (as defined by RAPP 1998) is influenced by the action of several hundred different compounds on the sensory organs.

The aim of this study was to compare the odour and flavour quality of two varieties, Rhine Riesling and Chardonnay, represented by regional types obtained from the South Moravian Region with the results of analysing their volatile aroma compounds by SFE/CGC.

MATERIAL AND METHODS

WINE VARIETIES

The aroma composition of two cultivars of *Vitis vinifera* L., cv. Rhine Riesling and Chardonnay, from South Moravian Region was investigated during vintage 1997, 1998 and 1999. Vintage grape samples were collected at four ripening stages at the following grades: quality wine 12.9 °Brix, cabinet wine 20.1 °Brix, late harvest 21.8 °Brix, selection of grapes 24.5 °Brix. Before SFE, the samples of wine were preserved by NaN_3.

DIRECT SFE OF AQUEOUS MATRICES

The analytical methods based on the apparatus in which supercritical carbon dioxide is re-circulated through a water sample with 50 ml of wine. Supercritical fluid enters into the extraction column at its bottom. The equipment consists of a packed column, phase separator, liquid CO_2 reciprocal pump, aqueous phase pump, liquid phase restrictor and gaseous phase restrictor. The extracts were stored in the refrigerator until the analyses CGC-FID and/or CGC/MS were performed.

CGC ANALYSIS

Wine extracts were analysed by a gas chromatograph GC 8060 connected with mass spectrometer Trio 1000. The CGC was equipped with a fused silica capillary column DB-WAX (0.25 mm × 30 m, film thickness 0.25 µm, J & W Scientic). The mobile phase was helium at linear velocity of 35 cm/s (60°C). A sample of about 1.0 µl was injected through a (split/splitless) injector while the temperature interface was 250°C and 220°C, respectively. The oven temperature program started at 35°C and was held for 5 minutes. After that, the temperature was increased to 220°C at the rate of 1°C/min. The final temperature was held for 10 minutes.

Mass spectra were measured at scan mode under electron impact ionisation conditions (electron energy –70 eV). The mass range was 29–300 amu and the scan time was 1.0 s. The analyte collection method based on the mixing of supercritical effluent with overheated solvent vapours made recoveries over 80% of theoretical yield. The extraction step together with GC analysis was reproducible. RSDs 2–4% were found by triplicate extraction (VEJROSTA et al. 1998).

The compounds investigated were identified by comparison of measured spectra with the spectrum library (agreement better than 85%) or by comparison with retention time and spectrum of a standard compound when it was available.

A CSW Chromatography Workstation version 1.7 was used for acquiring and processing the data. Measured retention times and peak areas represented various samples of wines and regions.

SENSORY ANALYSIS

A panel of experts was employed for this study. The all-male panel was composed of 9 subjects from winemaking companies of the region. They had been trained to perform the profile descriptive analysis of wine. Before the panel started to work, quantitative descriptive methods were used to describe the two regional wines by the chromatographic analysis CGC-MS.

SAMPLE PRESENTATION

Wine samples (10°C, 700 mL) were presented in coded glasses and they had to be assessed for bouquet and flavour. The evaluation was carried out in isolated booths. For the sensory evaluation, the panellists had to open the coded flask.

PROFILE DESCRIPTIVE ANALYSIS OF WINE

The panellists were asked to rate the intensity of each descriptor on an unstructured scale 100 mm entered at the left end with low intensity and at the right end with high intensity. All of the data were analysed by Unistat programme software. The techniques used were cluster analysis and stepwise linear regression analysis.

RESULTS AND DISCUSSION

SELECTED PROFILE OF BOUQUET AND FLAVOUR OF WINE

Sensory analyses were performed on wines sampled from selected producers in the South Moravian Region in three vintages (Table 1). The samples originated from a must composition which could contain up to 5% of must made from a different variety than that given on the la-

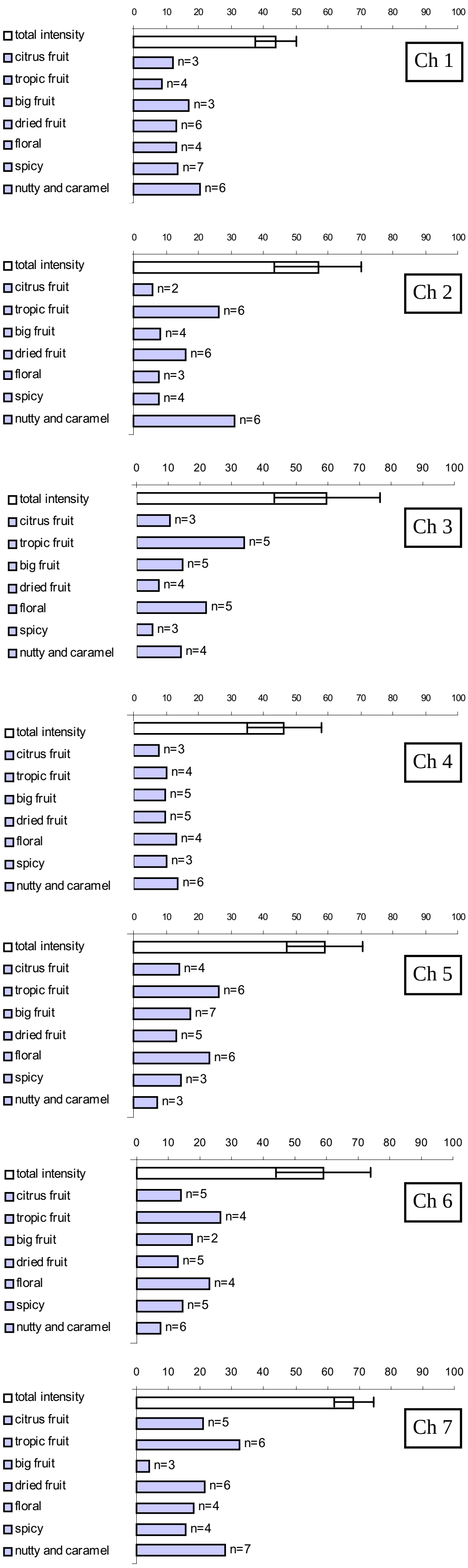

Fig. 1. Select profile of Chardonnay bouquet

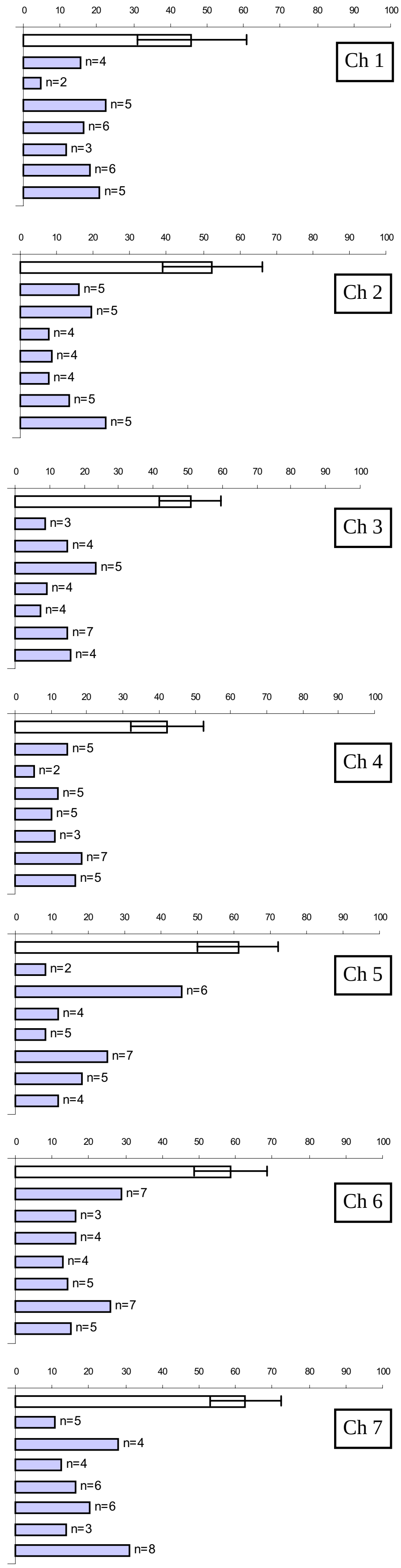

Fig. 2. Select profile of Chardonnay flavour

Table 1. List of wine samples

Label	Variety	Vintage	Quality level	Wine district
CH1	Chardonnay	99	quality wine	Velkopavlovická
CH2	Chardonnay	98	quality wine	Mikulovská
CH3	Chardonnay	99	late harvest	Velkopavlovická
CH4	Chardonnay	99	late harvest	Bzenecká
CH5	Chardonnay	99	selection of grapes	Mutěnická
CH6	Chardonnay	97	late harvest	Mikulovská
CH7	Chardonnay	99	selection of grapes	Podluží
CH8	Chardonnay	99	selection of grapes	Bzenecká
CH9	Chardonnay	99	selection of grapes	Mikulovská
RR1	Rhine Riesling	99	quality wine	Strážnická
RR2	Rhine Riesling	99	quality wine	Znojemská
RR3	Rhine Riesling	99	quality wine	Mutěnická
RR4	Rhine Riesling	99	cabinet wine	Strážnická
RR5	Rhine Riesling	99	late harvest	Mutěnická
RR6	Rhine Riesling	99	late harvest	Mutěnická
RR7	Rhine Riesling	99	late harvest	Mikulovská
RR8	Rhine Riesling	99	late harvest	Velkopavlovická
RR9	Rhine Riesling	99	late harvest	Znojemská
RR10	Rhine Riesling	97	late harvest	Mikulovská
RR11	Rhine Riesling	99	selection of grapes	Bzenecká

bel. Despite this fact, the discrimination of the variety wine by statistical analysis has a degree of plausibility.

The results (Figs. 1 and 2) showed 7 descriptors: **citrus fruit**, **tropic fruit, big fruit, dried fruit, floral, spicy, nutty and caramel**; the panellists were to differentiate the wine in bouquet and flavour.

The evaluating subjects (panellists) were capable of recognising bouquet best by the descriptors **tropic fruit,** and somewhat less **citrus fruit.** This is given by the number of subjects who could detect the presence of the descriptor in the given sample.

According to Fig. 1, no entry attains the maximum value ($n = 9$), but none of the descriptors was totally ignored in any sample. Therefore we cannot conclude that the profiles of the samples were significantly different. A similar situation arose in evaluating the flavour of the variety where the descriptors **tropic fruit, citrus fruit, nutty and caramel** and **spicy** were the ones most frequently used by the panellists. The least frequently chosen descriptor was **dried fruit.** The number of descriptors used in studies like this may be extended up to 16 (PRISER et al. 1997), which means that organoleptic properties are then rearranged into further subcategories of descriptors.

CLUSTER ANALYSIS OF CGC AND SENSORY VALUES

Cluster analysis was unable to distinguish the relative representation of components established by gas chromatography into two separated groups which would neatly demonstrate the character of a respective variety. This means that it was impossible to get two-group division corresponding to the division according to the variety (Fig. 3).

As the basis for drawing a dendrogram, the sum peak area of all substances found (87 compounds) in a given sample of wine was used. There were altogether 20 wines of both varieties (Rhine Riesling, Chardonnay). By naming each variety within the set of the original 20 samples and processing the data by cluster analysis, there arose two groups. In one group, Rhine Riesling is represented more frequently than in the other group (Fig. 4).

A similar distribution was provided by the cluster analysis of data obtained from sensory evaluation. In its uncorrected version, the distribution had both varieties in overlapping in clusters for bouquet: there was no homogeneous group of samples belonging to the same variety (Fig. 5).

After elimination of low distance values it is possible to separate three groups according to flavour where the statistics did not reflect the variety grouping (Fig. 6).

A comparison of the set of data obtained instrumentally by capillary gas chromatography and those obtained by sensory evaluation according to 7 descriptors for flavour and the same 7 descriptors for bouquet leads to the conclusion that the former (instrumental-analytical data) give more consistent results. This predictably corresponds to the higher financial and labour costs involved in laboratory measurements.

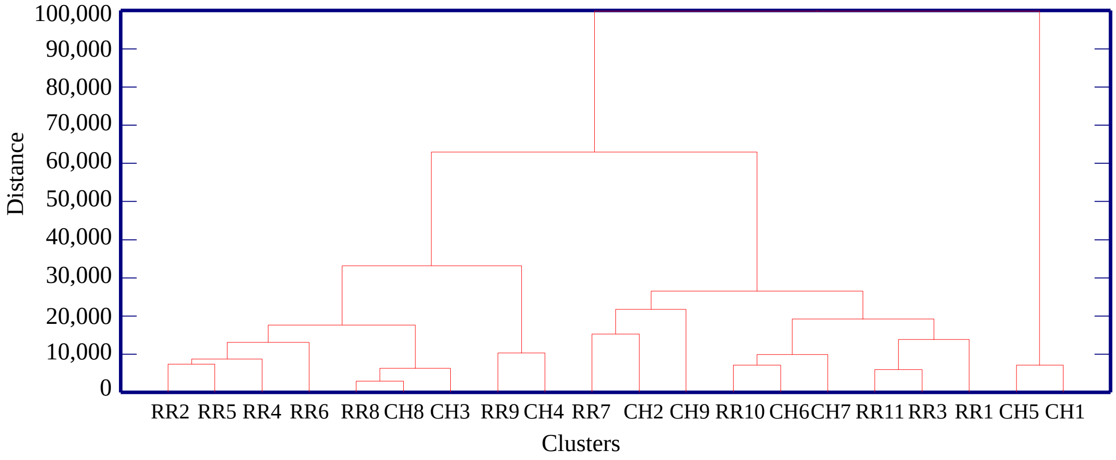

Fig. 3. Dendrogram of wines in dependence on relative occurrence of volatile substances

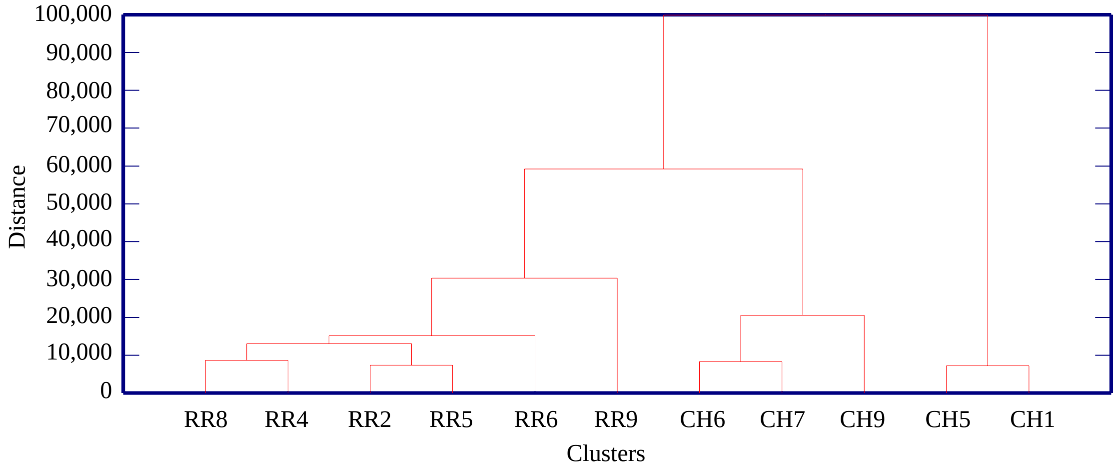

Fig. 4. Dendrogram of selected wines in dependence on relative occurrence of volatile substances

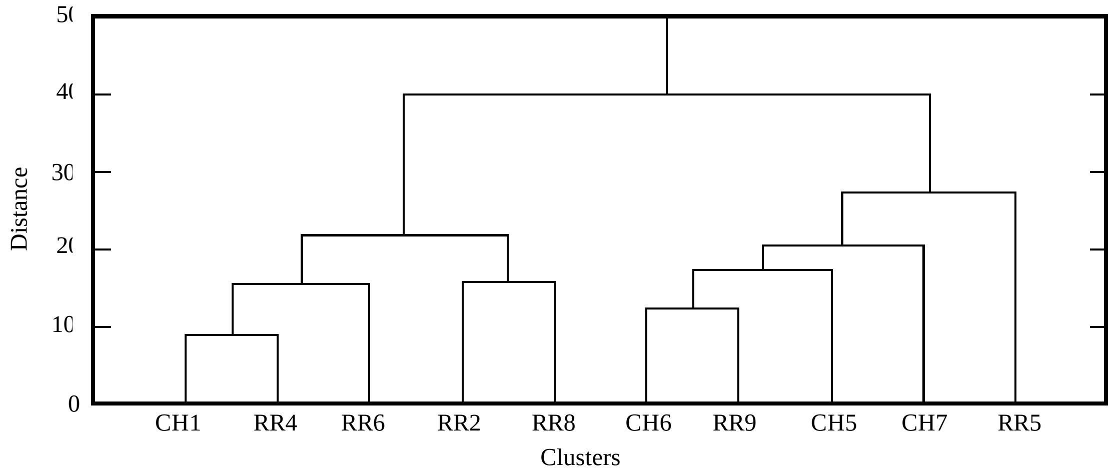

Fig. 5. Dendrogram of wines in dependence on sensory evaluation of bouquet

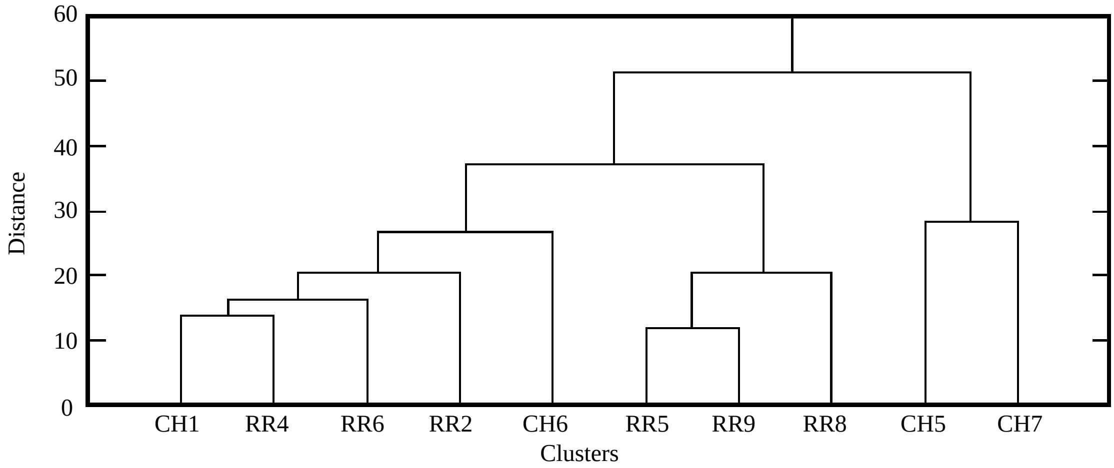

Fig. 6. Dendrogram of wines in dependence on sensory evaluation of flavour

FLAVOUR AND BOUQUET IN TERMS OF DESCRIPTORS VS. ANALYTICAL DATA OF CGC

Volatile substances of wine in the liquid phase which were established by the countercurrent SFE were regarded by human olfactory organs as bouquet. For the descriptor citrus, a multidimensional function has been proposed on the basis of stepwise linear regression. The function has the following general form:

$$y = a + b_1x_1 + b_2x_2 + b_kx_k$$

Seven variable components were selected by stepwise analysis from the total of 87 available ones. The seven selected variables seem to have contributed toward the sensory impression which could be designated as citrus bouquet. The coefficient of multiple correlation explains the effect of each component on the overall sensory evaluation (Table 4). Citrus flavour, for instance, results from

Table 2. Stepwise linear regression function of the dependence of citrus bouquet on volatile substances

Volatile compound SFE – variables (x_k)	Coefficient (b_k)	Multiple correlation	R-squared
Constant (a)	–12.8189	–	–
x_{C38}	0.0245	0.7460	0.5566
x_{C64}	0.0028	0.7953	0.6325
x_{C61}	–0.0230	0.8578	0.7358
x_{C30}	1.7755	0.8856	0.7843
x_{C2}	–0.1077	0.9256	0.8568
x_{C28}	–0.0014	0.9589	0.9195
x_{C68}	0.0532	0.9755	0.9515

Table 3. Stepwise linear regression function of the dependence of citrus flavour on volatile substances

Volatile compound SFE – variables (x_k)	Coefficient (b_k)	Multiple correlation	R-squared
Constant (a)	–8.2104	–	–
x_{C47}	–0.0860	0.5746	0.3301
x_{C71}	0.8149	0.7611	0.5793
x_{C87}	0.3634	0.8378	0.7020
x_{C58}	0.0007	0.9299	0.8647
x_{C27}	0.1124	0.9519	0.9061
x_{C86}	–0.0257	0.9709	0.9427
x_{C5}	–0.0211	0.9791	0.9587

Table 4. Average intensity of bouquet descriptors in wine and results of contingency analysis

Bouquet	Average intensity		Analysis of contingency	
	Chardonnay	Rhine Riesling	Chi-square	Significance
Citrus fruit	12.5	10.2	0.0083	0.9272
Tropic fruit	22.6	16.4	3.5549	0.0594
Big fruit	10.8	18.9	1.1169	0.2906
Dried fruit	13.7	12.3	1.6204	0.2030
Floral	16.3	18.2	0.5394	0.4627
Spicy	10.6	9.1	2.6278	0.1050
Nutty and caramel	18.9	10.8	8.9028	0.0028

Table 5. Average intensity of flavour descriptors in wine and results of contingency analysis

Flavour	Average intensity		Analysis of contingency	
	Chardonnay	Rhine Riesling	Chi-square	Significance
Citrus fruit	14.6	15.9	0.1697	0.6803
Tropic fruit	19.2	7.9	3.4266	0.0642
Big fruit	15.2	18.7	4.8855	0.0271
Dried fruit	11.6	9.9	2.8547	0.0911
Floral	13.9	14.6	0.2890	0.5909
Spicy	17.6	12.4	2.6299	0.1049
Nutty and caramel	19.2	14.0	0.4904	0.4837

several compounds from the analysed wine varieties (Rhine Riesling, Chardonnay). The descriptors suggested for the recognition of malolactic fermentation in Chardonnay were discussed by LAURENT et al. (1994). According to this author, in each case only one compound is responsible for the impression.

The present results after their statistical analysis seem to suggest a somewhat different conclusion: the first compound mentioned is admittedly prevalent in the creation of the sensory impression but it cannot be regarded as the one solely responsible for the integrity of the sensory descriptor (Tables 4 and 5). This result seems to be corroborated by the conclusion of LÓPEZ et al. (1999), who denies the existence of compounds which could be taken as characteristic of one variety only even though he achieves a one-to-one attribution of some descriptors to a definitive compound. In his opinion, the differences between varieties are quantitative rather than qualitative.

THE DIFFERENTIATION OF WINE VARIETIES ACCORDING TO THE FREQUENCIES OF BOUQUET AND FLAVOUR DESCRIPTORS

The descriptors for bouquet as well as those for flavour are assigned their respective average intensity (Tables 4 and 5). The significance of the average intensity has been obtained by contingency analysis. **Nutty and caramel** descriptors were significantly variety-dependent (Table 4). As far as the flavour was concerned, **big fruit** was shown to be statistically significant (Table 5).

The compounds established by SFE with a consecutive CGC analysis provided results that were processed by cluster analysis. The two varieties could be identified in two steps although both belong to middle-aromatic wines.

The contingency analysis found that out of the seven descriptors only **nutty and caramel** had a correlation with the bouquet of wine. On the other hand, in the case of flavour **big fruit** turned out to be the most decisive descriptor. Thus the relevant descriptors are not identical for flavour and bouquet.

It will be necessary to include even the process of wine maturation in the future research. The Chardonnay variety, when matured in bottles, seems to acquire a property that is associated with the descriptors **nutty and caramel** regarded by the panellists as the prevalent organoleptic characteristic in the bouquet of this wine variety.

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Složení těkavých látek vína a senzorické hodnocení vůně a aroma chuti dvou odrůd vína

ABSTRAKT: Byla srovnána analytická data získaná SFE/CGC se senzorickým hodnocením dvou odrůd vín Jižní Moravy. Senzorickou deskriptivní analýzou se prokázala velká variabilita organoleptických vlastností vín vedoucí k plnému vyjádření odrůdy vína. Klasterovou analýzou těkavých látek se rozlišily obě odrůdy vína. Naopak senzorická data se sedmi deskriptory pro buket a aroma chuti nebyla pro charakteristiku vína dostačující. Deskriptor pro citrusovou vůni byl popsán analyticky stanovenými látkami, jejichž statistický význam je doložený krokovou mnohonásobnou analýzou. Možnosti rozpoznání odrůdy vína senzorickými deskriptory jsou statisticky významné pouze pro ořechový a karamelový buket a vůni velkého ovoce pro aroma chuti.

Klíčová slova: víno; senzorická analýza; těkavé aromatické látky; superkritická extrakce/CGC

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